STUDIES ON ELECTRICAL AND DYE ADSORPTION PROPERTIES OF POLYANILINE-CHITIN NANOCOMPOSITES

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In recent years, one-dimensional (1-D) polyaniline (PANI) nanostructures, such as nanofibers, rods, and tubes, have been investigated for their potential to offer the combined benefits of low-dimensional materials and organic conductors. These one-dimensional polyaniline structures are typically synthesized through chemical or electrochemical polymerization. However, their limited solubility and mechanical properties present challenges for practical applications. To overcome this, conducting polymers blended with standard insulating polymers have been explored to combine the semiconducting/ metallic conductivity of polyaniline with the comparatively better mechanical properties of traditional polymers [*De Paoli et al., 1984*]. Processable PANI and its composites exhibit potential for a wide range of applications [*Cottevieille et al., 1999*]. Additionally, when blended with conducting polymers, the polymer matrix may gain protection from degradation, with polyaniline possibly providing an antioxidant effect in rubber mixtures [Helay et al., 1999], attributed to its ability to act as a radical scavenger [*Gizdavic-Nikolaidis et al., 2004*].

A vast amount of literature is available on conducting polymers and their composites. However, the present study is aimed at the PANI nanostructure and its composites, the literature review is limited to PANI and its composites and blends.

In the past two decades, researchers have made several attempts to improve the processability of PANI by synthesizing nanostructure counterparts as well as blending with other polymers so that its applications can be extended to new areas. McDiarmid and colleagues [Norris et al., 2000] successfully produced polyaniline/ poly (ethylene oxide) (PANI/PEO) composite fibers using the co-electrospinning method. Camphor sulfonic acid served as a dopant to enhance PANI's solubility. Microscopic analysis indicated that the resulting PANI/PEO composite fibers had diameters ranging from nanometers to micrometers. Despite their relatively large size, these fibers exhibited promising potential for

applications in electronic devices and sensors. By adjusting the PANI-to-PEO ratio, the composition of the composite fibers could be modified over a broad range. Mead and colleagues [*Wei et al., 2005; Wei et al., 2006*] developed polystyrene (PS)/PANI and polycarbonate (PC)/PANI nanocomposites featuring a core-sheath structure through the electrospinning of their blends. Transmission electron microscopy (TEM) images confirmed that the darker regions within the core represented the PANI phase, forming a continuous fibrillar structure, whereas the lighter regions corresponded to the PS or PC phases. The formation of this core-sheath structure in PS/PANI and PC/PANI nanocomposites is likely due to the high surface tension of PANI.

Araujo and co-workers [*Araujo et al., 2005*] developed composite nanofibers consisting of poly(methyl methacrylate) (PMMA) and polyaniline. HCl-doped polyaniline nanofibers (PANINF) with diameters between 100 and 150 nm were synthesized using an interfacial self-assembly method. Transparent PMMA/PANINF composite films with low filler concentrations were successfully produced by dispersing the nanostructures through sonication [*Araujo et al., 2005*]. Gharibi and co-workers [*Gharibi et al., 2006*] developed a gas diffusion electrode (GDE) incorporating a catalyst layer containing a mixture of Nafion and polyaniline nanofibers. Their results indicated that the presence of PANI within the catalyst layer reduced the polarization resistance of the electrode compared to those containing only Nafion. As a result, fuel cells utilizing PANI–Nafion electrodes are anticipated to achieve improved performance.

Fan and colleagues [*Fan et al., 2008*] fabricated a polyaniline/ polysulfone nanocomposite ultrafiltration membrane by filtering a polyaniline nanofiber dispersion onto a polysulfone membrane. SEM analysis confirmed a nanofiber layer on the surface, and the membrane exhibited a positive surface potential under acidic conditions due to PANI protonation. Kyoon and coworkers [*Kyoon et al., 2008*] created conducting

composite nanofibers from a polyaniline/poly(ethylene oxide) (PANI/PEO) blend mixed with multiwalled carbon nanotubes (MWNTs) via electrospinning. The PANI/PEO/MWNT nanofibers enhanced electrical conductivity, and an unexpected transition in conductivity was observed under a high electric field. Kim et al. introduced a hybrid polyaniline nanofiber–collagen composite material. The film remained conductive, though conductivity decreased with lower polyaniline content. Conductivity could be adjusted by de-doping in neutral water and re-doping in acid [*Kim et al., 2009*].

Kim and colleagues [*Kim et al., 2010*] developed a Nafion/polyaniline nanofiber composite for use as a flexible electrode in supercapacitors. The encapsulation of polyaniline nanofiber electrodes with Nafion led to enhanced energy density and cycle life. The composite exhibited a specific capacitance of 195 F g^{-1} and maintained stability over 10,000 charge-discharge cycles. Similarly, Banerjee and co-researchers [*Banerjee et al., 2010*] investigated the dielectric properties and charge transport characteristics of polyaniline nanofiber-reinforced PMMA nanocomposites. Chen and collaborators [*Chen et al., 2011*] studied the impact of chitosan on the interfacial polymerization of aniline. The chemical oxidation method was employed to synthesize nanostructured PANI at the interface between aqueous and organic phases. Their study highlighted that when the chitosan concentration in the aqueous phase was maintained below 1.5 wt%, polyaniline nanofibers were successfully formed. However, when chitosan content exceeded 2.5 wt%, polyaniline nanoparticles were primarily produced, indicating a critical threshold at approximately 2.0 wt% chitosan for controlling the nanostructure morphology of PANI.

Li and his team [*Li et al., 2011*] introduced a high-performance hydrogen sensor based on a polyaniline–chitosan nanocomposite. The composite sensor exhibited a superior response to hydrogen gas compared to conventional polyaniline nanofiber sensors, though its response polarity

was reversed. The study attributed this behavior to the composite structure, which comprised a conductive polyaniline nanofiber network coated with an insulating chitosan film. Additionally, chitosan served as a physicochemical filter, with its inter- and intra-chain spaces and functional groups effectively blocking larger or polar molecules. This property enabled the selective detection of hydrogen, the smallest and nonpolar gas molecule. Li and co-workers [*Li et al., 2011*] synthesized graphene nanosheets (GNS)/polyaniline nanofiber composites via in situ polymerization of aniline in perchloric acid ($HClO_4$). The polyaniline nanofibers uniformly coated the graphene nanosheets, significantly enhancing charge transfer reactions. The composite achieved a maximum specific capacitance of 1130 F g^{-1}. Furthermore, even after 1000 charge/discharge cycles, the specific capacitance retained 87% of its initial value, indicating high stability. Hopkins and team [*Hopkins et al., 2011*] investigated the structural properties of polyaniline nanofiber/polyimide composites using small-angle neutron scattering (SANS). Their findings confirmed that polyaniline nanofibers and polyimide were immiscible due to their conformational differences.

Recently, researchers have focused on biopolymers and polyaniline-based nanocomposites due to their biocompatibility and eco-friendly nature. Shahadat and colleagues [*Shahadat et al., 2022*] reviewed the advantages of chitosan and polyaniline-based nanocomposites. Disposal of nanocomposites after use doesn't cause any damage to the environment because of their biodegradable nature. And also noted that the combination of PANI and chitosan improves the applications of PANI in different fields such as wastewater treatment, biosensors, etc. Kong and co-researchers [*Kong et al., 2011*] directed their research toward the application of polyaniline composites for environmental remediation. The hydrophilic nature, biocompatibility, ease of availability, and nontoxicity make chitosan an interesting biopolymer for the adsorption process. Apart from that, chitosan is a low-cost biopolymer with a renewable nature. Antibacterial properties and fast adsorption kinetics are the

added advantages of such biopolymers [*Wong et al., 2004*]. However, disadvantages like very low conductivity, low surface area, pH sensitivity, and comparatively low porosity have limited the applications of chitin and chitosan on an industrial scale [*Lee et al., 2009*]. To overcome these disadvantages, one of the straightforward solutions is to prepare composites with suitable conducting polymers like PANI [*Veera et al., 2008*].

While PANI is a promising conductive polymer, its inherent brittleness and poor mechanical properties limit its practical applications. One of the methods to improve the properties of PANI is to synthesize its nanostructure counterparts. The synthesis of PANI nanostructures in several forms, such as nanosheets, nanofibers, nanorods, nanocoils, nanotubes, and nanoflowers, has been reported by various researchers. The properties of PANI depend on the surface area and porosity [Chen et al., 2005], as well as the dopants used, such as chloride, sulfate, etc.

Even though chitin and chitosan are both naturally occurring biopolymers, researchers have focused on chitosan because of its comparative ease of solubility in acidic medium. However, the advantage of chitin over chitosan is its superior mechanical strength and structural stability, making it more suitable for applications requiring high durability, such as packaging materials and structural composites. Additionally, chitin has a lower solubility in acidic conditions compared to chitosan, which can be advantageous in environments where controlled degradation or water resistance is required. Chitin also retains a higher degree of acetylation, contributing to its improved biocompatibility and potential for use in biomedical applications. Unlike chitosan, which requires chemical or enzymatic deacetylation for its production, chitin can be used in its natural form, reducing processing costs and environmental impact. These advantages make chitin a promising material.

After an extensive review of polyaniline-biopolymer blends and composites, it was determined that synthesizing polyaniline nanofibers and incorporating them into the chitin matrix would be a promising approach. To the best of our knowledge, no research has been carried out to incorporate PANI nanofibers in the chitin matrix so far.

In the present work, an attempt is made to incorporate polyaniline nanofibers in the chitin matrix to prepare a novel chitin-polyaniline nanocomposite with the following objectives:

1. Synthesis of polyaniline nanofibers and characterization.
2. Preparation of polyaniline-chitin nanocomposites and characterization
3. Effect of HCl doping on dielectric and ac conductivity of polyaniline-chitin nanocomposites.
4. Adsorption studies of indigo carmine (an anionic dye) on polyaniline-chitin nanocomposites.
5. The effect of doping on the adsorption capability of polyaniline-chitin nanocomposites.

The thesis is divided into six chapters. A brief description of the contents included in each chapter is given below.

In **Chapter 1**, a brief introduction to the invention of intrinsically conductive polymers (ICPs) and their advantages in different fields has been discussed. The chapter primarily focused on polyaniline, a well-known intrinsically conducting polymer, polyaniline nanostructures, and polyaniline-based composites. The different popular methods for synthesizing polyaniline nanostructures, template & non-template synthesis, are discussed in detail in the chapter and followed by their application in different fields. Finally, a review of literature relevant to the polyaniline-based composites, motivation, and objective of the present work has also been highlighted in this chapter.

In **Chapter 2**, details of the materials and chemicals used for the present study are described. Experimental techniques adopted for the characterization of samples include UV-Vis spectrophotometry, Fourier transform infrared spectroscopy (FTIR spectroscopy), X-ray diffractometry, Differential scanning calorimetry (DSC), Thermal gravimetric analysis (TGA), Field Emission Scanning Electron Microscopy (FESEM), and Mechanical studies. The theory behind the dielectric permittivity and ac conductivity is also discussed. A brief explanation of the principles of the techniques used and analysis procedures is given in this chapter.

The synthesis of polyaniline nanofibers by rapid mixing reaction, the preparation of polyaniline-chitin nanocomposites, and their characterization are discussed in **Chapter 3**. All the prepared composites were characterized by different techniques, namely UV Vis spectroscopy, FTIR spectroscopy, X-ray diffraction analysis, Thermal analysis, Mechanical properties analysis, and morphological studies. A redshift in the UV–Vis absorption spectra was observed with increasing polyaniline nanofiber content, indicating increasing conductivity. The optical energy gap was found to decrease with increasing polyaniline nanofiber emeraldine base in the sample. The UV Vis spectra of 1M HCl doped composite film show that the conductivity increases with the increase of reaction time. There were no observable peak shifts in the FTIR spectra of polyaniline-chitin nanocomposite compared to pure chitin, which indicates the formation of physical composites. The XRD analysis shows the overall decrease in crystallinity with increasing polyaniline in the composites. Increased water absorption of composites compared to pure chitin as seen in the DSC thermogram, may be due to the presence of hydrophilic LiCl pseudo-doped polyaniline nanofibers in the composites as well as increased porosity in the composites. TGA and DTG studies show that the overall thermal stability of prepared composites is higher than that of constituent polymers. The mechanical studies show that the tensile strength of the composite films decreases with increasing

polyaniline nanofiber emeraldine base (PANINF EB) content in the composite, which may be due to the formation of globular aggregates of PANINF EB or the generation of micro-cracks in the composite films caused by the uneven distribution of polyaniline nanofiber. The FESEM Micrograph shows that synthesized polyaniline has nanofiber morphology with an average diameter of 50 nm. FESEM micrograph of the recovered polyaniline nanofiber emeraldine base from its dispersion verifies that there were no visible morphological changes for polyaniline nanofiber in its dispersed state. The surface morphology of pure chitin and composites shows a smooth surface for pure chitin and a porous surface for the composites. The composite exceeding 20 wt% of polyaniline nanofiber content showed microcracks in the FESEM micrograph.

The effect of polyaniline nanofiber content in the composites along with the doping effect on the dielectric behavior, ac conductivity, and Complex impedance of prepared polyaniline-chitin nanocomposites have been discussed in **Chapter 4**. A monotonic decrease in the dielectric permittivity with increasing frequency has been observed for both the undoped and doped composites. At higher frequencies, dielectric permittivity attains constant values. It has been also observed that the frequency-dependency of undoped polyaniline-chitin nanocomposites (CPNC) decreases with increasing wt% of polyaniline nanofiber in the composite (10 wt% and 20 wt%). However, a reverse trend is observed after doping with 1M HCl. The same nature is observed in the case of dielectric loss and loss tangent (tanδ). This is because of the difference in the dielectric relaxation process that occurs within the composite material in the presence of undoped and doped polyaniline nanofiber. The dielectric relaxation within the composite reduces with the amount of undoped polyaniline nanofiber and hence there will be reduced energy dissipation in the composite, leading to decreased tanδ values. However, doping introduces free charge carriers in the composite and increases energy dissipation, leading to higher dielectric loss and tanδ values. A decrease in the dielectric permittivity with increasing temperature is

observed for doped composites (dCPNC) due to increased mobility of charge carriers. Temperature-independent dielectric behaviour is seen for undoped (CPNC) samples.

The ac conductivity of undoped and doped polyaniline-chitin nanocomposites (CPNC & dCPNC) shows frequency-dependent behaviour and is aligned with Jonscher's universal power law. Beyond 5 wt% of undoped polyaniline, CPNC shows a fall in the conductivity, which may be due to Maxwell–Wanger–Sillars polarization (MWS) or phase separation in the composites or reduced mobility of charge carriers or the combined effect of all of the phenomena occur in the CPNC10 and CPNC20 (Composites having 10 wt% & 20 wt% of polyaniline nanofiber respectively). For dCPNC, enhanced conductivity has been observed in the entire range of frequencies when compared with CPNC due to doping. Doping increases the number of polarons and bipolarons, thereby increasing the density of free carriers inside the composite materials. Change in the value of frequency exponent, 's', at various temperatures for CPNC and dCPNC gives an idea about the conduction mechanism present in the composites. A monotonous increase in the exponents, 's, with the increasing temperature is observed for dCPNC, and thus it was correlated to the Non-overlapping small polaron tunnelling (SPT) model, and Quantum mechanical tunnelling (QMT) was well obeyed with the exponent, s values observed for CPNC. Cole-Cole plot or Nyquist plot shows that the impedance of CPNC is much higher when compared to dCPNC. It has also been noted that the presence of undoped polyaniline nanofibers increases the impedance of the composites over 5 wt% polyaniline content in the composite due to the reduced mobility of charge carriers in the CPNC. At the same time, a monotonous decrease in the impedance and thereby reduced relation time have been observed in the dCPNC with increasing polyaniline nanofiber content. This is due to the doped polyaniline nanofibers in the composites, which can enhance the mobility of charge carriers. With increasing temperature, both CPNC and dCPNC show an increase in the bulk resistance and thereby decreased

conductivity. It can be attributed to the effects of thermally activated charge carrier scattering and increased interfacial polarization in the composite materials during temperature upswing.

The adsorption and desorption studies for the prepared polyaniline-chitin nanocomposites are discussed in **Chapter 5**. The adsorption capacity of dCPNC was observed to increase with the percentage of polyaniline nanofiber emeraldine salt in the composites. dCPNC20 (Composites having 20 wt% of polyaniline nanofiber) shows the highest indigo carmine (IC) dye adsorption. The dye adsorption capacities of dCPNC20, CPNC20, and CPNC20T (CPNC20 treated with 0.5M ammonium hydroxide) were in the order of dCPNC20>CPNC20>CPNC20T. The values of isotherm parameters for the adsorption of IC dye on the dCPNC20, CPNC20, and CPNC20T indicate that the adsorption follows the Langmuir isotherm model for all three composite adsorbents studied. The highest adsorption efficiency was obtained for dCPNC20 (Q_m= 30.13 $mg.g^{-1}$) when compared to CPNC20 (Q_m= 21.30 $mg.g^{-1}$), and CPNC20T (Q_m= 19.63 $mg.g^{-1}$). Isotherm parameters calculated for the indigo carmine dye (IC) adsorption on the composite support a favorable adsorption process. Adsorption kinetic data indicate that the adsorption of IC dye on all three composite adsorbents follows pseudo-second-order kinetics rather than pseudo-first-order kinetics. Thermodynamic parameters obtained for the adsorption process show that the adsorption of IC dye on dCPNC20 is spontaneous, whereas the adsorption process on the CPNC20 and CPNC20T is non-spontaneous. For all three cases, the adsorption process was exothermic, and entropy was observed to be decreasing. Desorption efficiencies of the composite adsorbents dCPNC20, CPNC20, and CPNC20T were obtained as 82.25%, 93.38%, and 97.20%, respectively, for the first cycle and 78.09%, 88.88%, and 93.60%, respectively, for the third cycle. The decrease in desorption efficiencies with increasing cycles was negligible for three cycles. This opens a new opportunity for reusing prepared composite adsorbents in multiple adsorption-desorption cycles, enhancing their practical application in anionic dye removal processes.

The major conclusions drawn from the present studies and the scope for the future study are given in **Chapter 6**.

List of publications:

1. Praveen, P., Pattabi, M., Pattabi, R. M., & Rao, V. (2019). Dielectric behavior and AC conductivity of low-loaded polyaniline emeraldine base-biopolymer nanocomposite. *AIP Conference Proceedings, 2162*, 020030.

2. Praveen, P., & Rao, V. (2017). Facile preparation and characterization of chitin-polyaniline nanocomposite. *International Journal of Advance Research in Science and Engineering, 6*(8), 1316–1321.

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2. Praveen, P., & Rao, V. (2017, August 20). Facile preparation and characterization of chitin-polyaniline nanocomposite. *5th International Conference on Emerging Trends in Engineering, Technology, Science and Management (ICETETSM-17)*, Institution of Electronics and Telecommunication Engineers, Ganganagar, Bengaluru, Karnataka, India.

3. Praveen, P., & Rao, V. (2014, December 19–20). Chitin/polyaniline nanocomposite: Preparation and characterization. *International Conference on Polymer Composites (ICPC-2014)*, Department of Mechanical Engineering, National Institute of Technology, Surathkal, Karnataka, India.

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